Swami Ramanand Teerth Marathwada University, Nanded, Maharashtra, India

(UGC Recognized, NAAC Re-accredited 'B++' grade State University)

"Design and Development of Composite Semiconductor Metal Oxide Films for Detection of Toxic Gases"

Research Proposal Submitted
In Fulfillment of Registration to
Ph.D. Programme

In the Subject
Physics Faculty of Science & Technology

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Introduction

Nowadays, gas sensor monitoring systems are needed for small, inexpensive, highly sensitive gas sensors that protect the environment and industrial applications. For example, placed a gas sensor not only protects unhealthy emission of gases but also saves us from dangerous situations. Although some techniques of monitoring gas sensors are expensive and lack portability. Hence, quick and smart monitoring systems are required for detection of various toxic gases. Protection of environmental pollutions and industrial safety is required as a global issue now days.

A gas sensor is an analytical device that uses a chemical responsive layer to recognize a change in the environment and convert that information into an electrical signal. In general, sensors are defined as the devices which receive signals and convert them into electrical forms which are further intended for electronics devices. It is often assumed that these chemical sensors are interfaces between the chemical world and electronics equipment's. Sensors differ from transducers in that they convert received signals into electrical signals while transducers only convert one form of energy into another.

Detection of harmful and toxic gases is important to became a point of necessity to control the environmental balance **[1-2].** Industrial waste gases from chemical industries, fuel combustion such as CO, NO_2, H_2S and NH_3 are of typical focus among researchers over the years. Rapid change in industrialization have tremendously increased environmental pollution. Due to growing industrialization, constant environmental monitoring and pollution control become essential **[3-6]**. The developments of portable gas sensors that are able to detect various gases with good sensing performance will significantly impact of our daily life **[7].** Excess concentration of these harmful gases presents in environment possess serious threats to human health **[8].**

In this era of growing demand for environmental safety and energy preservation, the necessity for control over combustible gases from a lot of the reactions right from domestic applications to small scale for industrial, automotive, aerospace and marine had risen. The challenging part of the Sensors are expected to work in extreme conditions like high operating temperature, high humidity, high pressure, high radiation levels, aggressive media which can be explosive, toxic, corrosive etc **[9,10].** Due to increase in rate of gas emissions has negative impact on human and environmental health. Protect from such toxic and hazardous gases we need to develop gas sensors. Gas Sensor is an device which senses the various gases that are in physical quantity in the one form and it converts into the another

form. Gas sensor is an electronic device which senses the amount of gas and produces the output information signal in the form of electrical energy signal.

Ideal sensor is a conceptual sensing element of active layer with supporting instrumentation showing quick response and fast recovery, highly selective and sensitive to a particular gas in the mixture of various gases even with higher concentrations, infinitely longer life, smart and intelligent, no power dissipation, negligible cost, etc. In actual practice, such sensors having ideal characteristics are not possible to fabricate. However, keeping this view in mind, the full attention has been paid and devoted to achieve the better characteristics of fabricated sensors.

Need of Gas sensors:

Metal oxides based thin film chemical sensors are used in a variety of applications, including toxic gas detection. These metal oxides have been used for over long periods of time as inexpensive gas sensor devices. These metal oxide sensors are extremely helpful for protecting purposes such as food industry gas detection, environmental gas detection; space applications, industrial hazards gas detection; biomedical gas detection etc. These sensors can be used at room temperature with low cost and high sensitivity and low power smart sensor applications.

Presently, the atmospheric pollution has become a global issue. Gases from auto and industrial exhausts are polluting the environment. The gases like CO, H_2, Cl_2, CO_2, NO_2, NH_3, CH_4, H_2S, etc. have to be controlled for the healthy survival of the human beings. In order to detect measure and control these gases, one should know the how much number of gases present in the environment and also various type of gases present in the atmosphere. Thus, the need to monitor and control these gases has laid to the research and development of a wide variety of gas sensors using different materials and technologies. The importance of environmental gas monitoring is well observing, many studies have focused on the development of suitable gas sensing materials **[11-13].**

Researchers are trying to fabricate gas sensors with fast response, long-term stability, high selectivity, and greater sensitivity **[14].** There is an important role to control and monitor these harmful gases, considering a high risk of damage to assets and human beings if a leak occurs. There are many gases which can be toxic for human beings. Accordingly, there is a need of gas sensors to continuous and effective detection of these toxic gases to avoid

predominantly the above said dangers. An ideal gas sensor should possess the following sensing properties **[15]:**

- High Sensitivity
- High Selectivity
- Long term Stability
- Response time
- Reversibility
- Faster response time and good recovery time.
- Simple principle of operation
- Easy to handle.
- Simple fabrication techniques.
- Working Temperature.
- Detection limit
- Hysteresis.

Literature Review

In earlier studies, we found that pure or doped WO_3 is a favourable material for the detection of various gases. Tungsten oxide (WO_3) is an n-type semiconductor and resistance increase in the presence of oxidizing gases, such as ozone and nitric oxide, and decreases in presence of reducing gases like hydrogen sulphide.

Pure WO_3 and metal tungstate thick films have outstanding sensitive properties toward nitrogen oxides at low temperatures. Tungsten oxide (WO_3) is an n-type semiconductor with band gap of about 2.6-2.8eV [16]. Tungsten has many oxidation states i.e,.2,3,4,5 and 6. Tungsten trioxide exhibits a cubic perovskite like structure based on the corner sharing of WO_6 octahedral, with the O atoms at the corner (centre) of each octahedron [17]. In 1967 first report on WO_3 for gas sensing was published by Shaver [18]. It has been found that WO_3 can be used for detecting a variety of toxic gases such as hydrogen (H_2)**[19],** methane (CH_4), ammonia (NH_3)**[20-23],** carbon monoxide (CO) **[24],** nitric oxide (NO) **[25,26],** oxygen (O_2), hydrogen sulphide (H_2S)**[27,28],** nitrogen dioxide (NO_2) **[29],** ethanol (C_2H_5OH), ozone (O_3), trimethylamine ($(CH_3)_3N$), sulphur dioxide (SO_2), and chlorine (Cl_2).

Review of In_2O_3 gas sensors for the detection of Ozone (O_3) **[30]**, Hydrogen (H_2), Carbon monoxide (CO) **[31,32],** Hydrogen Sulphide (H_2S) **[33,34]**, Nitrogen dioxide (NO_2) **[35]**, Nitric oxide(NO) **[36].** In addition In_2O_3 based gas sensors are used for the detection of alcohol vapour [37], ammonia [38,39]. ZnO is an gas sensing material has been studied by

many researchers because of its chemical sensitivity to different adsorbed gases, high chemical stability, non-toxicity and low cost. Review indicates that ZnO gas sensors used for the detection of H_2,**[40],** CO, NO_2**[41],** NH_3, and H_2S gases respectively.

From the literature study, it is clear that WO_3, ZnO and In_2O_3 based gas sensors are of high interest at the moment because of their good sensing properties to various gases. Different methods have been used to improve gas sensitivity of tungsten trioxide, zinc oxide and indium oxides including modification of morphology and microstructure (i.e., grain size, film thickness, phase) by using different deposition techniques, or by doping with different metals.

National / International Status:

Nisha, R Cochin university of science and technology, India has worked on the development of semiconductor metal oxide gas sensors for detection of gases. Avnish Kumar Arora Department of Chemistry, Bijnor (U.P.), India. Has been studied metal oxides/mixed metal oxides have wide application in gas sensors. Departement of electronics and Instrumentation, SRM Institute of Science and Technology, Kattankulathur, Tamil Nadu, India Jeyapaul sam Jebakumar and Asokan V. Juliet worked on doped and undoped tin oxide gas sensors for detection of pollutant gas. Prof. Rajaram mane, Gas sensing and energy devices loboratory,SRTMU Nanded, India working in this field from last decades.

Gas sensing characteristics of doped and undoped tungsten oxide thin films was studied by Tuquabo Tesfamichael , Andrea Ponzoni , Mohammed Ahsan, Guido Faglia in Sensor laboratory, University of Brescia & IDASC-CNR, Via Valotti 9, 25133 Brescia, Italy and Queensland University of Technology, 2 George Street, Brisbane, 4000 QLD, Australia. Study shows that the performance of these films in sensing four gases (H2, NH3, NO2 and N2O) in the concentration range 2–10,000 ppm at operating temperatures of 150–280 ◦C has been investigated. Doping of the tungsten oxide film significantly increases the base conductance of the pure film but decreases the gas sensing response.

Yanmin Wang studied ZnO nanorods have been widely used to detect low-concentration gases due to its range of conductance variability, response toward both oxidative and reductive gases, and highly sensitive and selective properties. Technical University, Bld. Stefan cel Mare, 168, 2004 Chisinau, Moldova and d Department of Electronics, University of Barcelona, Marti i Franqus 1, 08028 Barcelona, Spain, Matti A. Maki-Jaskari, V. Golovanov, have been studied Polycrystalline In_2O_3 films deposited by spray pyrolysis were analyzed by using XRD, HRTEM and XPS methods, he found the

response of In_2O_3-based gas sensors to oxidizing gases is limited by diffusion type processes.

Objective:

India is the 7th most environmentally insecure country over the world. Among India's about 6% of children and 2% of adults have Asthma due to air pollution. On NDTV report, it has been published that, Delhi is the most air polluted city in the world. Industrial and vehicular emissions lot of toxic and hazardous gases expelled in atmosphere as a result pollution is increasing all over the world and these causes the various problems to living beings.

At present we need to develop a device which detects the ominous atmosphere and provide protective comfortable life to society. We need to develop such device towards the development various gas sensors. Due to wide range of applications and the scope for practical development of the gas sensors we are working in this field. **[42,43]**.

Objective of the study includes sensing properties of Thick/Thin film sensors based on pure and copper doped tungsten oxide. Study the synthesis of pure and doped Nano crystalline powders and gas sensors were fabricated from prepared powders. The structural properties, compositional analysis and morphology of obtained materials are characterized with SEM, EDS, Raman Spectroscopy, XRD. Sensors evaluated at different operating temperatures under concentration of target gas. Once operating temperature found, we are going to study different concentration of target gas.

As compared to other semiconductor metal oxides like ZnO, WO_3, SnO_2, In_2O_3 is little bit novel for gas sensing studies. In this work we are going to study the gas sensing properties of pure and doped indium oxide gas sensors to test the toxic and hazardous gases present in the environment. Objective is to fabricating, synthesis and characterization of thin film gas sensors based on pure and indium doped ZnO fabricate on glass substrate. The structural properties, chemical and compositional properties, characterizations of prepared sensors. We are going to study performance of pure and doped sensors for test gases. This work will investigate to achieve the above-mentioned objective:

1. To synthesize Metal oxides like In_2O_3, ZnO, WO_3, Sb_2O_3, CrO_3 for preparation of active material for Gas sensor.
2. Prepare Pure and Composite material with different Metal Oxide to fabricate Gas sensor.
3. Prepare thin film by using active material with different deposition Method.
2. Structural and morphology analysis characterize synthesized thin films and nanostructure

using XRD, UV-Vis Spectroscopy, Raman spectroscopy, FTIR, TGA-DTA, SEM-EDS, XPS, etc.

4. To study the Sensing characteristics for Hydrogen Sulphide (H_2S) and Methane (CH_4) Gas of Prepared Pure and Composite thin films.
5. To propose optimize sensor based on the results and material properties.

Experimental Methodology:

1. Sensor testing setup

Sensor characterisation is an important feature to identify the various aspects of performance, of the gas sensor in order to optimise its use. We are going to make testing setup. Testing at the development stage must also determine the parameters, which need to be included in the performance tests essential for any sensors.

2. Methods of Measurement

The response of a gas sensor is tested by measuring the resistance or conductance of the sensor element in air and in the presence of a known amount of the gas **[44].**

3. Active Layer Deposition Technology

Various deposition technologies have been tested effectively. Processing techniques should be able to afford the desired oxide composition with specific doping and the minimum number of processing steps. Film processing techniques are grouped in two main categories: thin-film deposition processes such as sputtering, evaporation (i.e. physical vapour deposition – PVD) and chemical vapour deposition (CVD), for thicknesses in the range 0.005-2μm, and thick-film deposition processes such as screen printing, drop coating and tape casting for thicknesses greater than 10μm. Thermal spraying can be used to deposit coatings of metals, ceramics and cerements that are thicker than ~50μm. Below are summarised the different processing methods used for synthesising gas sensor films **[45].**

The nanocrystalline powders were synthesized by precipitation technique from aqueous solutions further we are doing some processes and fabricating gas sensor.

4. Thin Film Technology

Many chemical and physical methods of thin-film deposition are available. Chemical vapour deposition (CVD) involves the flow of a gas with diffused reactants over a hot surface **[46]** . Physical vapour deposition (PVD) involves the direct impingement of particles on the substrate surfaces. The most important feature of PVD techniques is that the transport of vapours from the source to the substrate takes place by physical means. There are different

methods of PVD **[47]**. The sol-gel technique consists of a system going from a liquid sol (colloidal suspension of miniature solid particles in a liquid) to a viscous gel in which the suspended particles are organised in a loose, but definite three dimensional arrangement **[48]**.

Fig. Sensor Fabrication techniques.

Characterization

Characterization of a material is an important step after its synthesis because it gives useful parameters in determining the properties of polymers. Chemical characterization consists of determination of oxidation states. Physical characterization consists of spectroscopic analysis viz. Fourier Transform Infrared and UV-visible spectroscopy, TGA-DTA analyses, etc.

The spectroscopic characterization of the polymer samples is usually carried out by FIR and UV-Vis spectroscopy. UV-Vis spectroscopy gives the energy band gap and defect states, while FTIR spectroscopy identifies and confirms the structure and presence of various linkages in polymer.

Different measurement techniques to characterize the structural, magnetic, dielectric andelectrical properties of the specimens can be employed.

1. X-ray powder diffraction (XRD) generally, polymers are considered to be amorphous in nature. However, it was observed that the XRD pattern of conducting polymer differs because of uncertainty in polymeric chains. It depends on synthesis route, solvent and ionized state.
2. A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
3. The morphology, particle size and shape are revealed through field emission scanning microscopy (FE-SEM).Field emission scanning electron microscopy (FESEM) provides topographical and elemental information at magnifications of 10x to 300,000x, with virtually unlimited depth of field. Compared with convention scanning electron microscopy.

4. For thin crystalline samples, TEM produces an image that consists of a pattern of dots in the case of a single crystal, or a series of rings in the case of polycrystalline or amorphous materials. TEM images are used for determination of deposition of nanoparticles in conducting polymers, a histogram of particle size distribution of the nanoparticles can also be found out.
5. FTIR provide information on the basis of chemical composition and physical state of the whole sample So all these above mentioned are the characterization techniques are very useful forknowing the material structure and properties of the material.

Year wise tentative Plan of Action:

Year 1:

- Review of Semiconductor Metal oxide material like In_2O_3, ZnO, WO_3, Sb_2O_3, CrO_3 etc and Selection of Metal Oxide materials for preparation of sensor thick film.
- Synthesis of Materials.
- Preparation of Active material from Selected Metal Oxide.

Yaer 2:

- Fabrication of thin film of active material with different thin film methods.
- Characterization of active prepared material for structural and Morphological analysis includes x-ray diffraction(XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), optical spectroscopy, Raman spectroscopy, x-ray photoelectron spectroscopy (XPS) thermogravimetric analysis(TGA), etc.
- structural and Morphological analysis of active material using various data of XRD,TEM,SEM,AFM,OS,RS,XPS and TGA

Year 3:

- Performance of fabricated sensor for Hydrogen Sulphide (H_2S) and Methane (CH_4) Gas.
- To study Sensitivity , Selectivity, Stability , Lower detection Limit(LDL), Reproducibility, Response and Recovery Time for Prepared composite sensor.
- From the structural , Morphological and Gas sensor response characteristics Optimize best sensor for gas detection.

Scope of Work

Detection of various gases and determining their compositional parameter has constantly been on the increase in recent years. The demand for better environmental stability and safety has increased research activities of solid-state gas sensors. Gas sensors using metal oxide semiconductors have been the subject of further investigations for more than three decades, In more recent research, the interests shifted to some other promising metal oxides, with interesting properties as gas sensing materials. Using metal oxides has several advantages, features such as simplicity in device structure, low cost for fabrication, robustness in practical applications and

adaptability to a wide variety of reducing or oxidising gases. Gas sensors based on nano metal oxide materials are the focus of current research activity of sensor designers. In this present work we are trying to address these problems. For detection of pollutant, toxic and hazardous gases we need to develop the metal oxide gas sensor we have lot of chance to design doped and pure semiconductor metal oxide gas sensor used to detect the gases.

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Consent Letter from Ph.D. Supervisor

To
The Assistant Registrar,
PG Section (Ph.D.Unit)
Swami Ramanand Teerth Marathwada University,
Nanded-431606

Subject: Consent Letter to be a supervisor for Mr. Hemant Kishanrao Suryawanshi as a research Student under Ph.D. Programme.

Dear Sir

With reference to the above-mentioned subject, I wish to inform you that I am willing to accept Mr. Hemant Kishanrao Suryawanshi as my PhD student, and for guiding his research work leading to PhD degree of Swami Ramanand Teerth Marathwada University, Nanded. I will guide him for the entire duration of his research work and will supervise him work throughout the research process.

Following is the proposed title of his Ph.D. research work:

"Design and Development of Composite Semiconductor Metal Oxide Films for Detection of Toxic Gases"

Thanking you

Yours Sincerely

Date:15/09/2022
Place: Parbhani

.

Name of Research Scholar:

Mr. Suryawanshi Hemant Kishanrao

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Subject: Physics

Research Topic: **"Design and Development of Composite Semiconductor Metal Oxide Films for Detection of Toxic Gases"**

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